

## Corrosion Resistance of Reinforcing Steel in Concrete Using Natural Fibers Treated with Used Engine Oil

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### Abstract

The addition of natural fibers in the elaboration of concrete pastes has increased as an innovative alternative for the development of more ecological and environmentally friendly constructions. The objective of this research is to incorporate natural fiber residues from palm leaves and mango stone impregnated with used engine oil (UEO) in the cement matrix to improve the mechanical and electrochemical properties of reinforced concrete. Samples with fiber percentages of 0.2% and 0.4% with respect to the weight of the sand with a length of 10 mm were fabricated. Their properties, such as workability, air content, porosity, and compressive and flexural strength, were analyzed. To understand the corrosion rate of the steel bars, electrochemical techniques of corrosion potential, electrochemical noise, linear polarization resistance, and electrochemical impedance spectroscopy were applied to cubic samples exposed in a 3% sodium chloride saline environment for 365 days. The experimental results showed a positive effect on the corrosion phenomenon with the UEO and mango fiber treatment, decreasing the corrosion rate due to the formation of a protective film at the steel/concrete interface.

*Keywords:* Concrete; Natural Fibers; Corrosion; Used Engine Oil; Surface Treatment; Electrochemical.

## 1. Introduction

Concrete is a material used in construction due to its excellent mechanical properties in compression, and combined with reinforcing steel, the material acquires high tensile strengths. The cement paste passivates the steel, protecting it from corrosion agents due to its high alkalinity, which promotes a protective oxide layer that separates it from highly aggressive environments. However, corrosion of steel reinforcement and other embedded metals is the main cause of poor performance and durability problems in reinforced concrete (RC) structures [1]. The passive layer protecting the rebar is easily destroyed by carbonation and/or the presence of chlorides. Corrosion products occupy a larger volume than the original; internal steel corrosion products promoting tensile stresses will accumulate in the concrete [2]. After the passive layer is destroyed, a visible sign of corrosion appears on the surface of the structure, e.g., cracks, rust stains, concrete spalling, etc. [3]. To reduce the effects of corrosion, the need to obtain synthetic, natural, or waste materials as alternatives to conventional reinforced concrete has been sought [4].

The advantage of using natural waste as an alternative is its positive impact on the environment, which can be obtained at a low cost using traditional knowledge and practices [5–7]. How these natural materials are included in the concrete is through fibers such as jute [8], bamboo [9], coconut [10], palm [11], sugarcane bagasse [12], and others,

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which have several advantages, including low density, high flexural strength, flexibility, and high elastic modulus [13]. These fibers consist of cellulose micro-fibrils suspended in a matrix of amorphous lignin/hemicellulose composition, which gives the natural fibers their chemical composition [14]. The introduction of new types of vegetable fibers to concrete has been investigated as it has a great perspective and potential in the construction sector. Vegetable fibers from palm leaves and mango stones are innovative agricultural residues that can be used in adequate quantities as concrete reinforcement, although they present durability problems. Water absorption is one of the main problems of natural fiber composites, which reduces their mechanical properties and dimensional stability, limiting their use in various applications [15–17].

Zhan et al. [18] explored the effect of natural jute fibers on compressive strength, and the results are critical because natural fibers swell by absorbing water (during casting and their lifetime), creating spaces between the fibers and the matrix. Shah et al. [19] observed that increasing the concentration of natural fiber decreased the settlement values in all specimens. These reported decreases in workability are the result of moisture absorption by hydrophilic natural fibers [20]. Numerous researchers have incorporated chemical or physical treatments into natural fibers to protect them from water absorption and avoid disadvantages such as fiber/cement biocompatibility [21, 22]. Used engine oil (UEO), generated by industrial activity and transportation around the world, generates great pollution because it is discharged specifically into groundwater and soil, generating high levels of carbon dioxide [23, 24]. This material can be added as a natural fiber treatment because it is a hydrophobic material, does not require a manufacturing process, and is an attractive option for the elimination of OEU. Juarez Alvarado et al. [6] used hydrophobic substances: linseed oil, linseed oil + natural resin, kerosene, kerosene + natural resin, wood sealant, and creosote as surface treatments for the lechuguilla fibers to reduce the absorption of water, concluding that the protective treatment with kerosene reduced the absorption capacity of the natural fiber.

Yousif et al. [25] studied the treatment of kenaf fibers immersed in 4 solutions: water, salt water, diesel, and engine oil, and obtained the conclusion that oil and diesel had a lower absorption rate of 20–30% compared to the untreated fiber of 90%, although it reduced the tensile strength. Wong et al. [26] evaluated the effects of fiber treatment on absorption in motor oils and petroleum, and their results showed that vegetable fibers have increased oil absorption, which reduces water absorption. Ramli et. al. [27] evaluated coconut fibers incorporated in concrete with a percentage of silica fume as a binder and reported that, for more severe climates such as seawater chlorides, two factors influence the chloride penetration of concrete: the severity of exposure to the chloride source and the permeability of the specimens. However, the impact of the use of natural fibers treated with UEO on the corrosion of cementitious elements is still not clearly known.

The mechanical properties of concrete with natural fibers are variable; however, the scenarios and aggressive environments are still not clearly known. This work will take an innovative approach and provide further insight into the current literature on steel corrosion phenomena, so this research study describes the use of two natural fibers (mango stone and palm leaf) treated superficially with used engine oil (UEO) and used as reinforcements in concrete to improve mechanical and electrochemical properties such as compressive strength, flexural strength, and corrosion rate resistance.

## 2. Materials and Methods

### 2.1. Materials

Portland cement CPC 30 R was used for concrete sample preparation; according to the international ASTM C150 standard [28], river sand was used as fine aggregate with a 4.75 mm maximum particle size, crushed gravel (19 mm nominal size) as coarse aggregate, tap water, and natural fibers. The general methodology of the work is shown in the flowchart in Figure 1.

### 2.2. Fibers

Two types of fibers were used in the concrete: mango fibers (CMF) and palm fibers (CPF). Mango stone residues are considered as wastes, and solitary hermaphrodite Mexican Sabal palm are considered important component sources [29], distributed across the USA, Mexico, and Central America down to Venezuela and Colombia [30]. Both materials were cleaned, eliminating polluting residues, and afterward were dehydrated at room temperature for three days. The hollow tubular structure of the fibers also reduces their volume and density and makes them lighter [31], having different values as shown in Table 1.

**Table 1. Fiber density**

Fiber	Density (g/cm <sup>3</sup> )	Fiber Type	Reference
Mango	0.91-1.08	Woody	[32]
Palm leaf	0.5-0.8	Leaf	[11]

Short fibers were cut manually with dimensions of 10 mm in length and 3-4 mm in width, and 0.1 mm in thickness and presented in Figure 2.

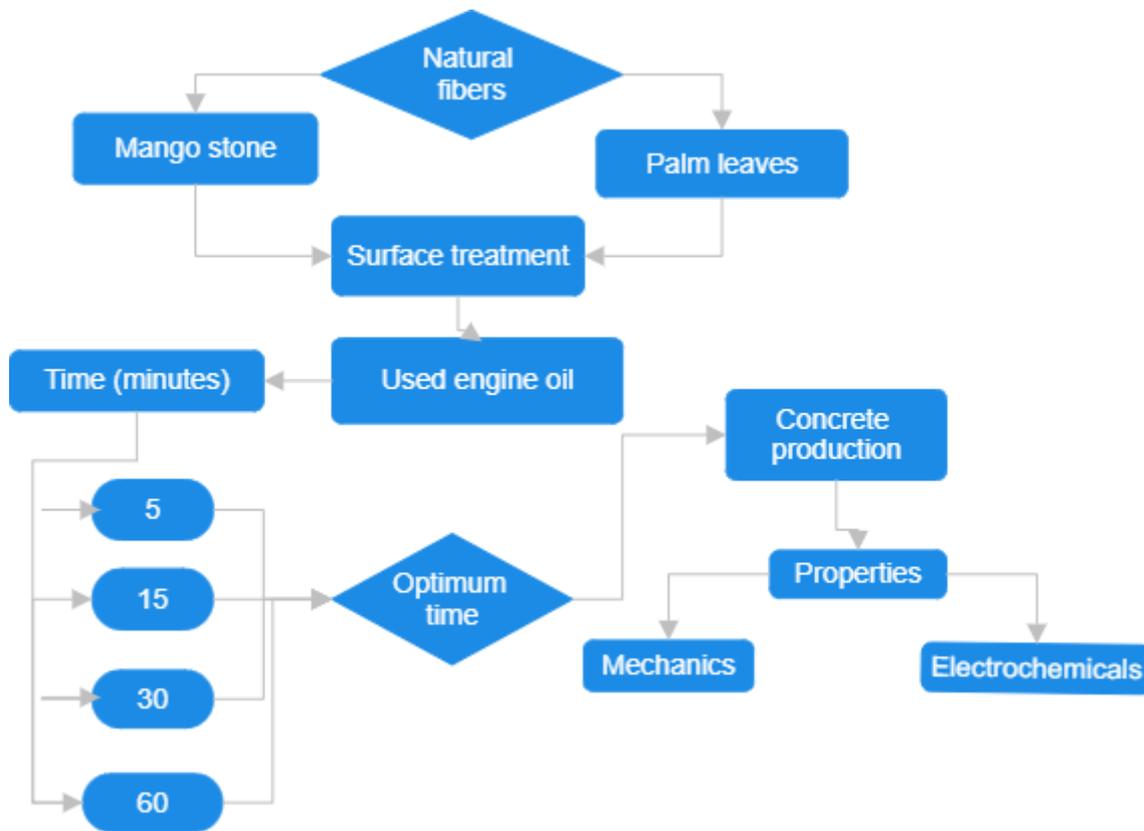


Figure 1. Flowchart of the experimental methodology

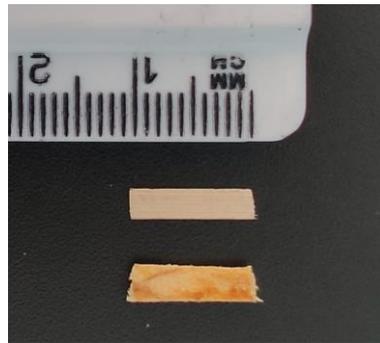


Figure 2. Measurements of natural fibers

### 2.3. Fibers Treatment

Fiber chemical treatment alters superficial energy and structural characteristics through the shallow cellular walls surfaces modification, without deteriorating its chemical composition [33]. The hydrophobic characteristic substance was used, being easy to obtain, recycled, and economical, with the purpose of reducing high water absorption, giving protection at high alkaline concrete. The UEO chemical properties are shown in Table 2.

Table 2. Used motor oil chemical properties

Chemical composition	Used engine oil (%)	Reference
Sulfur (SO <sub>3</sub> )	31- 37	[34, 35]
Chloride (Cl <sup>-</sup> )	14.9-15.9	[34, 35]
Lead content (mg/kg)	3.6	[34]
Zinc (ZnO)	17.7-19.2	[34, 35]
Nitrite (Ni)	5.5	[34]
P <sub>2</sub> O <sub>5</sub>	8.95	[35]
Others	4.12	[35]

Natural fibers surface treatment (TNF) using capillary impregnation using 30 CMF fiber or 50 CPF fiber strips. Afterwards, they were immersed in UEO at 70 °C temperature for 5, 15, 30, and 60 minutes as shown in Figure 3. These times are considered to obtain the best conditions to penetrate fibers completely and less natural fibers water absorption.

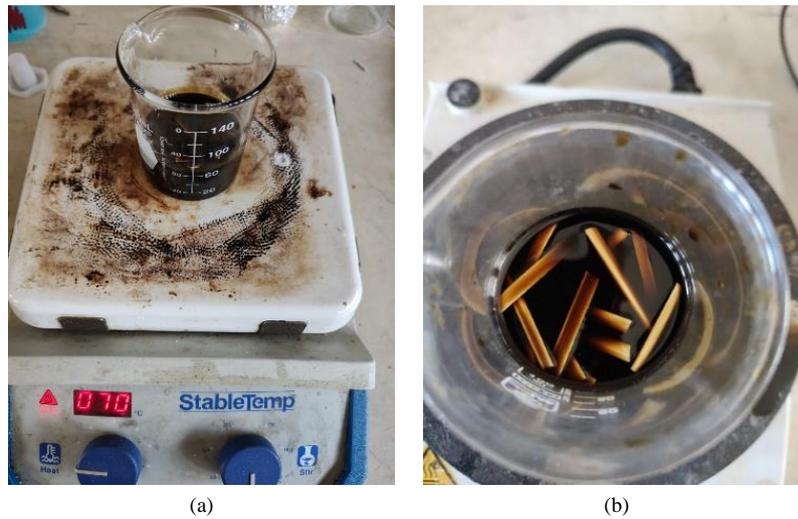


Figure 3. a) Surface impregnation of the fibers b) Fiber saturation

Afterward, humidity absorption was determined, by saturating fibers in distilled water for 24 hours, removing them, and cleansed with an absorbent cloth to retire UEO excess until obtaining dry superficial fibers, as seen in Figure 4. Then fibers were weighed on a 0.0001 g precision Denver Instrument electronic scale. The samples were dried again at 70 °C for one hour and weighed again.



Figure 4. Surface drying of treated fibers

For the calculation of water absorption percent (Wa %) Equation 1 was used, where W is the saturated water humidity weight and Wo dried weight at 70 °C [36-38]:

$$Wa\% = \frac{W - W_0}{W_0} \times 100 \tag{1}$$

Table 3 presents the natural fiber superficial treatment average absorption percent in 5, 15, 30, and 60 minutes immersed in UEO. In general, it can be appreciated the different percentages of mango and palm natural fibers, noticing fibers without treatment absorb a greater water quantity due to the presence of waxes and threads in the external layer from untreated fibers absorbing more from the solutions [25]. The water absorption diminished in treated fibers at 15 minutes immersed in the hydrophobic medium; that is, the treatment time presents a better response to the absorption resistance material as compared with untreated fiber absorption, therefore this immersion time was selected for concrete preparation.

Table 3. Absorption average percent for natural fibers

Fiber	Absorption in %				
	Untreated	Treatment (minutes)			
		5	15	30	60
CMF	39.24	34.69	31.24	29.84	29.92
CPF	56.91	37.71	37.64	45.64	50.33

## 2.4. Concrete Preparation

Table 4 presents the dosage for 80 liters of concrete using the absolute volumes method. A 300 kg/cm<sup>2</sup> (29.44 MPa) theoretical mix adding up fiber percents of 0.2 or 0.4 with respect to the sand weight, with 0.54 water/cement relation, without any additive.

**Table 4. Concrete proportions for 80 liters**

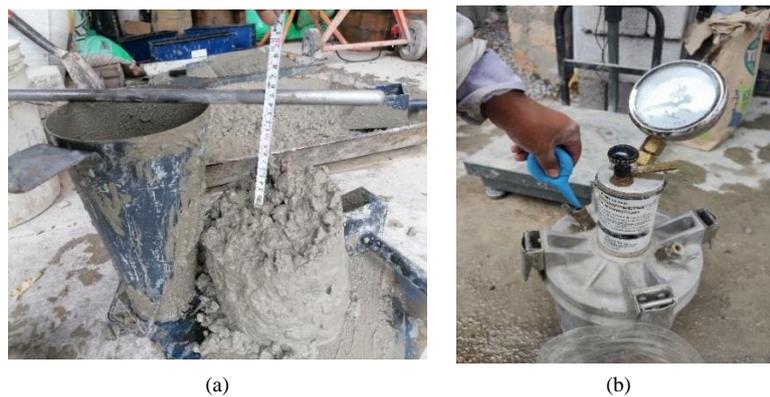
Material	C	0.2% fiber	0.4% fiber
Cement (Kg)	29.286	29.286	29.286
Water (l)	15.811	15.811	15.811
a/c	0.54	0.54	0.54
Sand (Kg)	52.049	51.947	51.842
Gravel (Kg)	83.811	83.811	83.811
Fiber (Kg)	0	0.104	0.208

Five mix designs were prepared, two mixtures of concrete with mango fibers (CMF,) in two percentages mentioned, and two mixtures of palm fiber (CPF) and control concrete (C). The nomenclature of the samples is specified in Table 5.

**Table 5. Identification for samples**

Material	Type of fiber	Percentage (%)	Nomenclature
	--	--	C
Cement	Mango	0.2	CMF-02
		0.4	CMF-04
	Palm	0.2	CPF-02
		0.4	CPF-04

The mixture was prepared as follows: first gravel, after a small quantity of water was poured. Then sand was added, then the cement, and finally the rest of the water incorporating the TNF. Physical tests such as air contained in the mixture, according to the standards [39, 40], as seen in Figure 5. Cylindrical specimens were prepared with standard dimensions of 15 cm diameter and 30 cm height [41] for 7 and 28 days of compression testing.



**Figure 5. a) Slump test b) Air content**

Samples were transported and kept in adequate conditions of humidity, and curing for 28 days. For flexural tests, concrete blocks 50 cm long, 15 cm wide, and 15 cm high were manufactured [41] for 28 days resistance test. A universal press with 120 tons maximum capacity was used for mechanical concrete tests. The standard procedure was used [42, 43] (load at third points) and flexural resistance was calculated using Equation 2:

$$f'f = \frac{PL}{bd^2} \quad (2)$$

where  $f'f$  is the flexural resistance,  $P$  is the applied load,  $L$  is the distance between supporting points,  $b$  is the width, and  $d$  is the average slope of the sample as shown in Figure 6.



Figure 6. Concrete flexural strength test

Cubic samples were made for the electrochemical tests, with dimensions of 12 cm length, 8 cm width, and 12 cm height as shown in Figure 7, and three low carbon steel rods were embedded. The steel rods were named working electrodes  $W_1$  and  $W_2$ , and a third rod was used as an auxiliary electrode according to the electrochemical cell proposed for the different measurement techniques. The coating of the paste with the steel bar was 3.5 cm, simulating a structural element, and Teflon tape was placed at the steel/paste interface in order to avoid contamination or any alteration in the measurements. The contact area of the rebar embedded in the concrete is 22.30 cm<sup>2</sup>. The specimens were transported to a place with adequate humidity conditions, curing for 28 days.

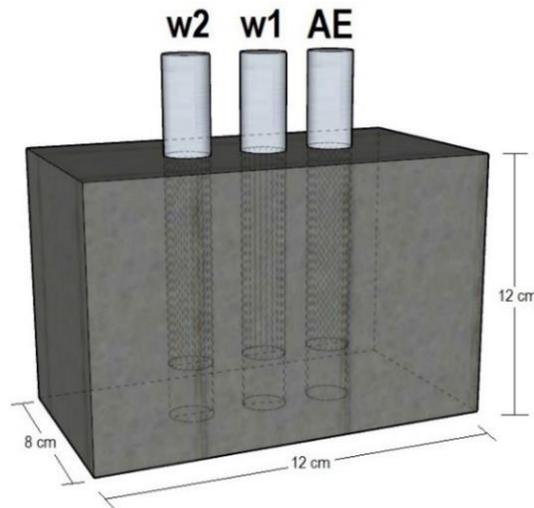


Figure 7. Sample dimension for electrochemical tests

### 3. Electrochemical Techniques

#### 3.1. Hall-Cell Open Circuit Potential

The  $E_{corr}$  corrosion potential, also known as half-cell potential (HCP), is the open circuit potential (OCP) of the rebar [44]. ASTM C876-09 [45] relates HCP to the probability of corrosion of reinforcing bars in concrete. With the values obtained, it is feasible to elaborate a diagnosis of the degree of corrosion risk of the concrete reinforcement [46]. The electrochemical cell for HCP monitoring was made with a saturated silver/silver chloride reference electrode (Ag/AgCl) and a working electrode W, both electrodes were connected to a multimeter. The measurement started 24 hours after the immersion of the specimens in the saline solution, the interpretation of the data is the average of the measurements of the 3 steel rods. Table 6 shows the parameters for interpreting the corrosion potential in reinforced concrete.

Table 6. Corrosion potential parameters  $E_{corr}$

Reference electrode Ag/AgCl (mV)	Corrosion risk (%)
less than -150	10% Corrosion probability
-150 to -300	Uncertain zone
more than -300	90% Corrosion probability

### 3.2. Electrochemical Noise

The method involves the measurement of small fluctuations in the current signals created between two nominally identical electrodes, and the voltage signals that are generated between the reference electrode and the working electrodes [47]. The electrochemical cell connection for the other electrochemical techniques is described in Figure 8, two working electrodes  $W_1$  and  $W_2$  were used as corrosive working elements, a reference electrode (RE) Ag/AgCl was used, and closing the circuit a third rod was used as auxiliary electrode (AE). A Gill AC-ACM Instruments device was used, and measurements of 1024 readings were taken at a standard rate of 1 reading/second.

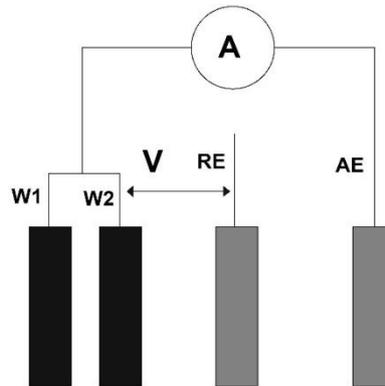


Figure 8. Electrochemical cell configuration

The electrochemical noise resistance ( $R_n$ ) was calculated with the quotient of the standard deviations of potential ( $\sigma_v$ ) over current ( $\sigma_i$ ).

$$R_n = \frac{\sigma_v}{\sigma_i} \tag{3}$$

Another statistical data that can be obtained with  $R_n$  measurements is the localization index, which is related to the type of corrosion that occurs: generalized (value close to 0) or localized (value close to 1) [48]. Table 7 shows the range of values for the different types of corrosion that occur in the steel bar.

Table 7. Range of current values for the type of corrosion

Location index values	type of corrosion on the steel surface
0.001 - 0.01	Generalized
0.01 - 0.1	Mixed
0.1 - 1	Localized
> 1	Start of pitting

The corrosion rate,  $V_{corr}$  represents the volumetric loss of metal per unit area and unit time, and is obtained from the corrosion current, through Faraday's law, and the density of the metal, for steel,  $1 \mu A/cm^2$  is equivalent to a corrosion rate of 0.0116 mm/year [49]:

$$V_{corr} \left( \frac{mm}{year} \right) = 0.116 * I_{corr} \left( \frac{\mu A}{cm^2} \right) \tag{3}$$

### 3.3. Linear Polarization Resistance

A Gill AC-ACM instruments potentiostat was used to measure the LPR technique, the layout of the electrochemical cell is described in the preceding section, a small amplitude of potential values of -50 mV and +50 mV were applied close to  $E_{corr}$ , by ASTM G-59 standard [50], and a sweep rate of 60 mV/min.

The polarization resistance  $R_p$  was calculated according to Equation 5, which defines the tangent of the polarization curve in the anodic and cathodic regions in the narrowest possible polarization range around the corrosion potential [51, 52]. To obtain  $I_{corr}$ , the polarization resistance ( $R_p$ ) is related to Stern-Geary, then the constant B depends on the anodic and cathodic Tafel slope constants, According to Equation 6, the guidelines for measuring  $I_{corr}$  adopt the values of B as 26 and 52 mV for active and passive steels, respectively [53, 54]:

$$R_p = \frac{dE}{di} \tag{4}$$

$$I_{corr} = \frac{bac}{2.303(ba+bc)} = \frac{B}{R_p} \tag{5}$$

According to Andrade & Alonso [49], a current density ratio  $I_{corr}$  can be obtained to estimate the degree of corrosion and durability of reinforced concrete structures, parameters as shown in Table 8.

**Table 8. Electrochemical parameters of  $I_{corr}$  on the service life of reinforced concrete**

$I_{corr}$ ( $\mu\text{A}/\text{cm}^2$ )	Degree of corrosion in the metal
< 0.1	Negligible
0.1 – 0.5	Moderate
0.5 – 1	High
> 1	Very High

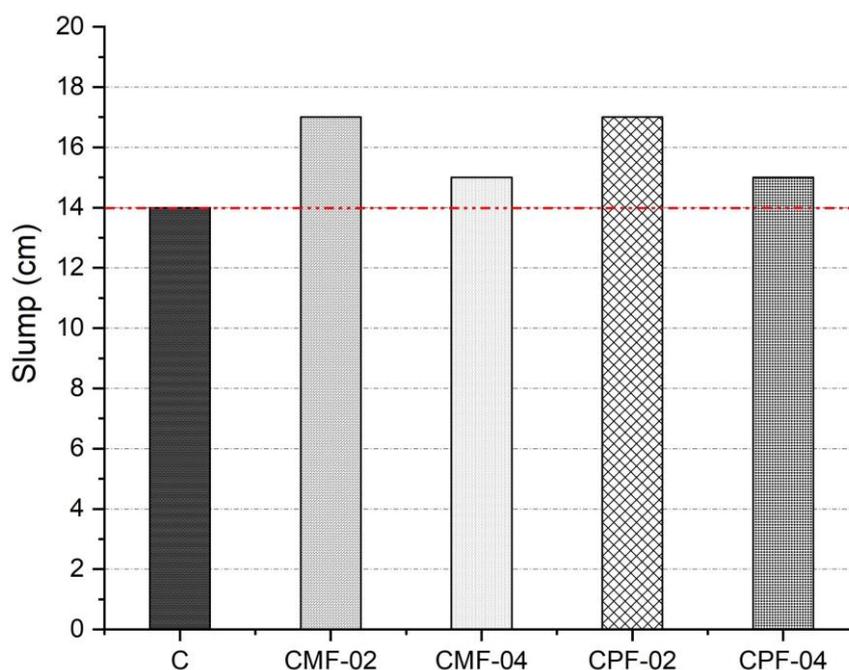
### 3.4. Electrochemical Impedance Spectroscopy

Electrochemical impedance spectroscopy (EIS) is a non-destructive testing method that uses low amplitude and variable frequency sweep alternating currents to study the properties of materials [55]. It can be considered an informative method because does not only measures the polarization resistance but also evaluates the physical processes within the concrete and at the steel/concrete interface [56]. The EIS measurement technique sweep was performed in a frequency range of 0.01 Hz (low) to 10000 Hz (high), and a sinusoidal perturbation of  $\pm 20$  mV was applied, with the support of potentiostat equipment attached to a computer and appropriate software.

## 4. Results and Discussion

### 4.1. Physical Properties of Fiber-Reinforced Concrete

The slump test is used to evaluate the consistency or workability of the concrete mix. The workability is an indicator of quality control of materials such as fine and coarse aggregates with the cementitious matrix [4]. Workability concrete results are shown in Figure 9 where the concrete control presents a 14 cm slump effect, and when adding treated natural fibers (TNF) an annealing increase due to the reduction in water quantity was obtained, and a consequence of UEO acting as a water reducer during the curing process, and the increase in a slump from high contents of  $\text{SO}_3$  in UEO [35, 57]. It was shown that the UEO particles act as a super-plasticizer in the concrete mix and these results are in agreement with other authors [58, 59]. The results for air content are shown in Figure 10, where it can be observed that the specimens with treated fibers increased the amount of voids in the cementitious matrix, which could indicate that the use of WEU caused a bad interaction between the paste and the fiber, i.e., the adhesion decreased drastically for samples CPF-02 and CPF-04 with air content values of 133% and 123% with respect to the control sample, showing that the increase of UEO absorption increases the air content.



**Figure 9. Slump variation in concrete with TNF**

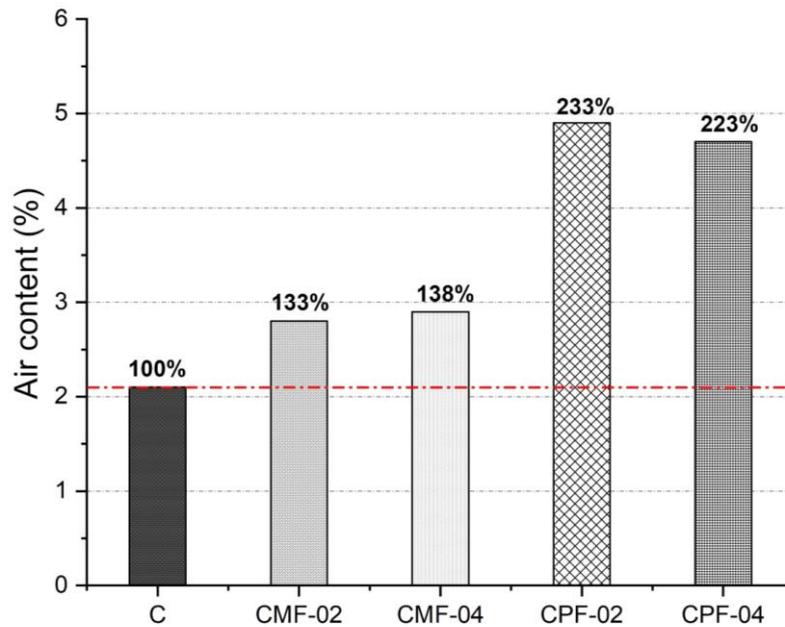


Figure 10. Air content in concrete with UEO-treated fibers

In the complex concrete microstructure, pores may be present in nanometers up to the macro scale [60], and porosity was obtained from air content results [61]. Figure 11 presents the concrete porosity results, where sample C shows 19% porosity, with an a/c relation of 0.54, being this result similar as reported by other authors [46, 62, 63]. All samples with TNF present more void content when compared to the control sample provoking an increase in permeability, directly affecting its compression resistance [64]. It can be appreciated that CPF-02 and CPF-04 samples present a higher percent porosity of 34% and 32%, possibly due to UEO directly affecting the interfacial zone between the aggregates and fibers matrices.

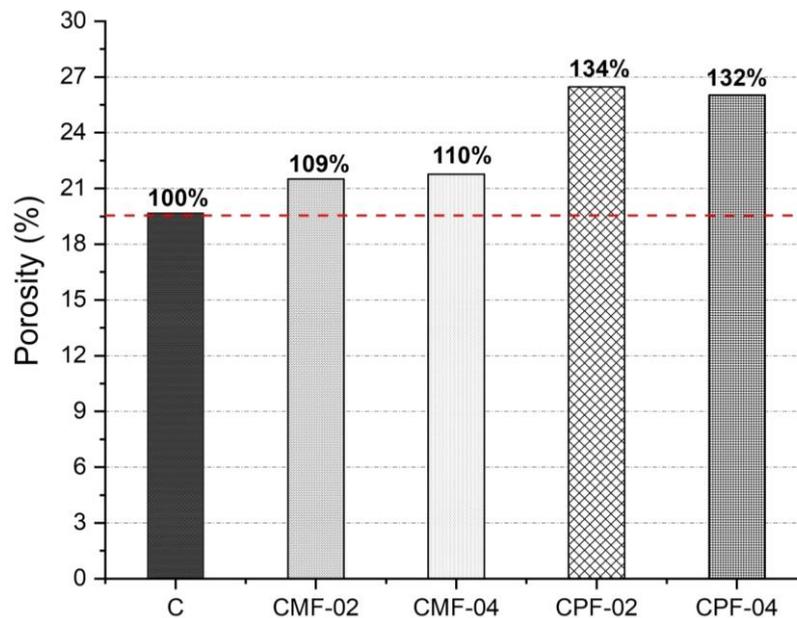


Figure 11. Percentage porosity of concrete with TNF

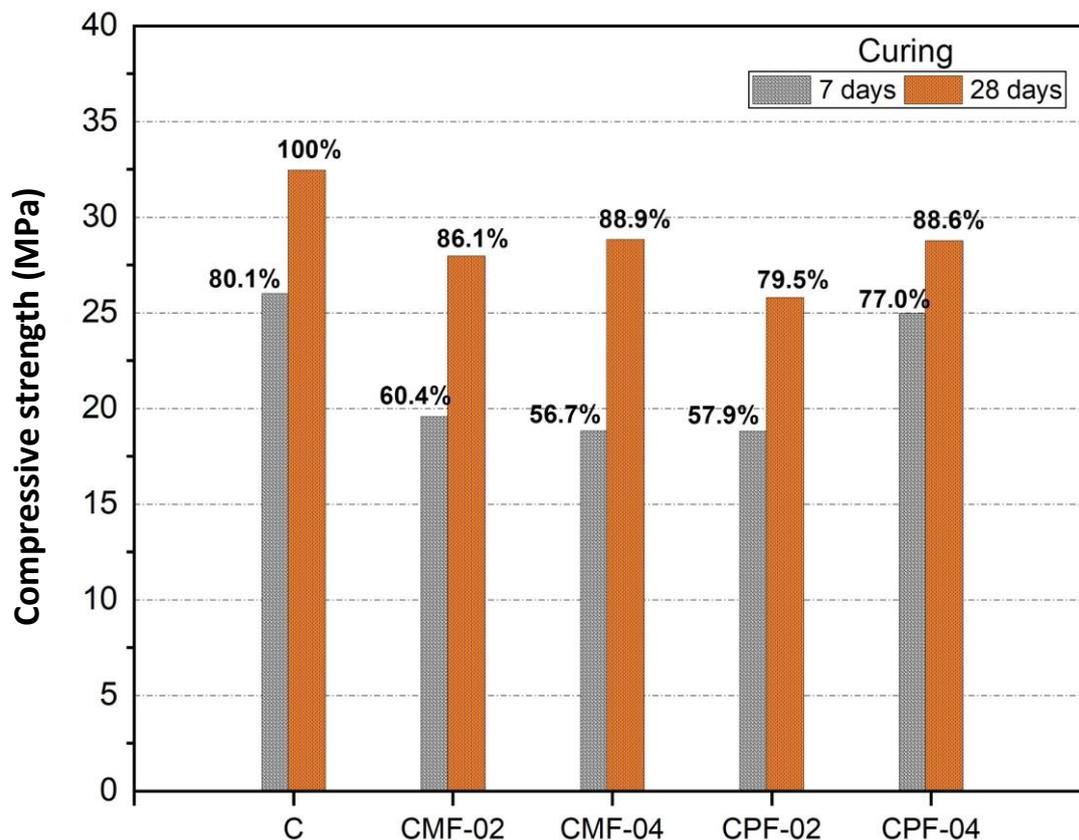
#### 4.2. Compressive Behavior of Concrete Specimens

Average compression resistance results obtained at 7 and 28 curing days are presented in Table 9. The control sample presents a higher compression resistance ( $F'_c$ ) as compared to the other samples, due to the presence of UEO from natural fibers; this is due to the increase in the interfacial transition zone (ITZ) surrounding the aggregates with the fiber. A greater ITZ presents a weaker structure as compared with the greater part of the paste being more fragile to mechanical loads [65]. In addition, a reduced level of chemical reactions is demonstrated in the presence of oil along with the weakening of cohesive forces [31].

**Table 9. Mechanical compression properties of concrete in its different dosages**

Material	F'c 28 days (MPa)						F'c 7 days (MPa)	F'c 28 days (MPa)
	7 days			14 days				
Age	N° 1	N°2	N°3	N° 1	N°2	N°3		
C	26.5	25.79	25.72	33.042	32.23	32.08	26.0	32.5
CMF-02	19.8	20.01	18.93	28.243	27.91	27.74	19.6	28.0
CMF-04	18.351	195.4	17.78	29.342	28.76	28.44	18.4	28.9
CPF-02	19.078	193.3	18.42	26.114	25.65	25.66	18.8	25.8
CPF-04	25.525	252.7	24.57	28.754	28.56	29.04	25.0	28.8

This decrease in the F'c is similar to the ones reported by different authors adding up a small percentage of UEO in the concrete paste [66-70]. The percentage decrease of F'c with oil impregnation in the natural fibers ranges from 11.1% to 20% with respect to sample C, as can be seen in Figure 12. At the early curing time of 7 days, it can be observed that samples CMF-04 and CPF-02 have a lower development in their mechanical properties as a consequence of the hydrophobic characteristic of the UEO. This causes a delayed reaction of Ca(OH)<sub>2</sub>, which has not yet been completely consumed for the production of calcium silicate hydrate (CSH); therefore, the bonding between the fiber and the matrix is poor [71].



**Figure 12. Variations of compressive strength during the curing process**

### 4.3. Flexural Behavior of Concrete Specimens

Average concrete flexural resistance (F'f) at 28 curing days is shown in Figure 13. The effects of the UEO on the flexural strength are negative, decreasing by 25% with respect to the control sample, and the incorporation of the fiber in the concrete paste does not prevent the propagation of cracks when the material is subjected to stresses. The Flexural strength of concrete decreases due to the weakened bond interface, the failure mode of the fibers is predominantly because of heavy metals present in the used oil engine, namely ZnO and P<sub>2</sub>O<sub>5</sub> [20, 58]. With the use of UEO and fibers, a decrease in the mechanical properties of concrete has been reported elsewhere [72-74].

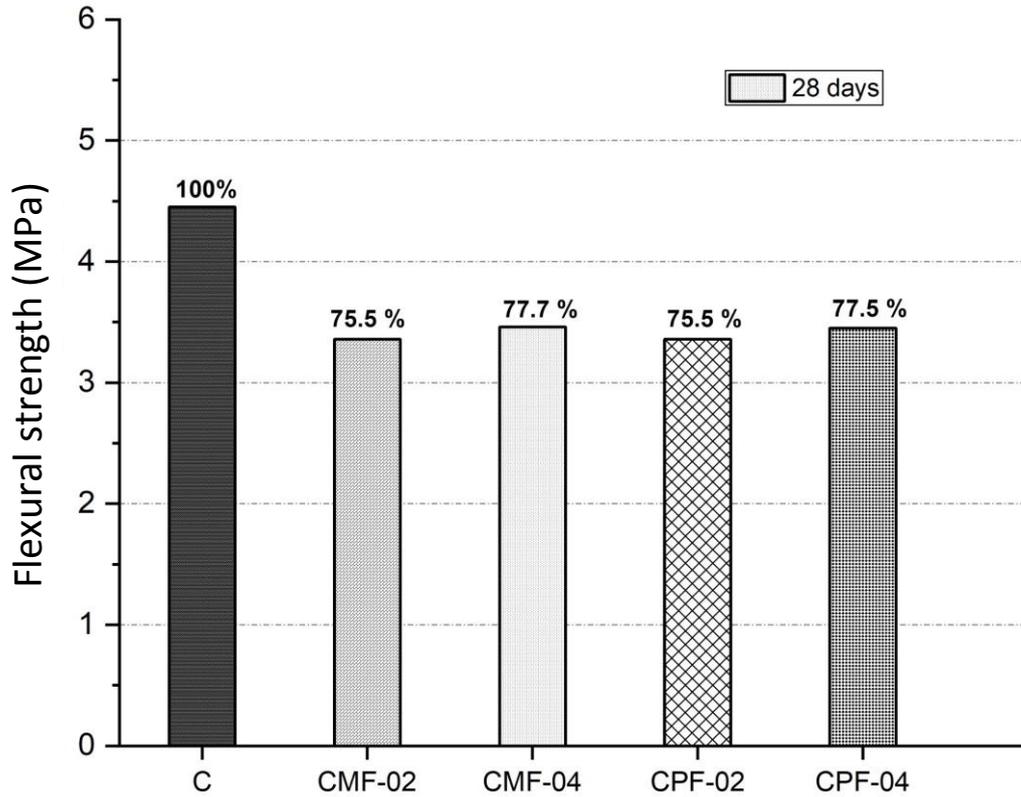


Figure 13. Variation of flexural strength of concrete with TNF, cured at 28 days

The results of the cracks in the flexural test are shown in Figure 14-a), as it can be generally noted that all specimens show a single crack passing through the center in the third of the beam. Specimen control shows a vertical crack characteristic of reinforced concrete; likewise, the specimens with natural fibers CPF-02 and CMF-02, show a brittle shear failure of diagonal type, as previously described the tensile strengths are low, the main cause is the null adhesion of the UEO treated fiber with the cement matrix as can be observed in Figure 14-b).

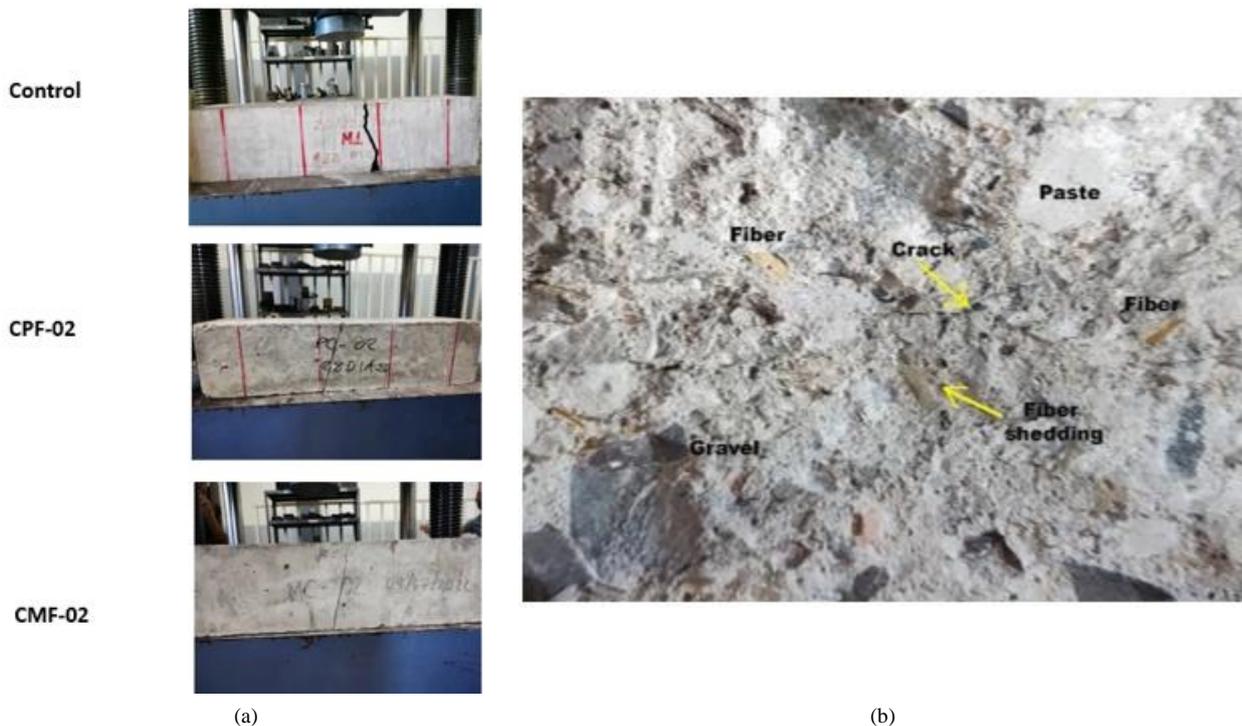


Figure 14. a) Fracture of specimens in flexural tests; b) Macro structure

#### 4.4. Interpretation of Corrosion Potential $E_{corr}$

During the corrosion process, an electric current flow between the cathodic and anodic sites through the concrete and this flow can be detected by measuring the potential drop across the concrete [75]. Therefore,  $E_{corr}$  corrosion potential measurement is a non-destructive method that detects these changes on the metal surface.

The corrosion potentials for the concrete samples with oil-treated fibers are shown in Figure 15. At the beginning of the monitoring, it is observed that the sample CMF-04, presents more positive  $E_{corr}$  values of -100 mV compared to the other samples, placing it in a zone of 10% probability of corrosion. This phenomenon indicates a good distribution of the fibers that delay the transport of fluids and chloride ions [34].

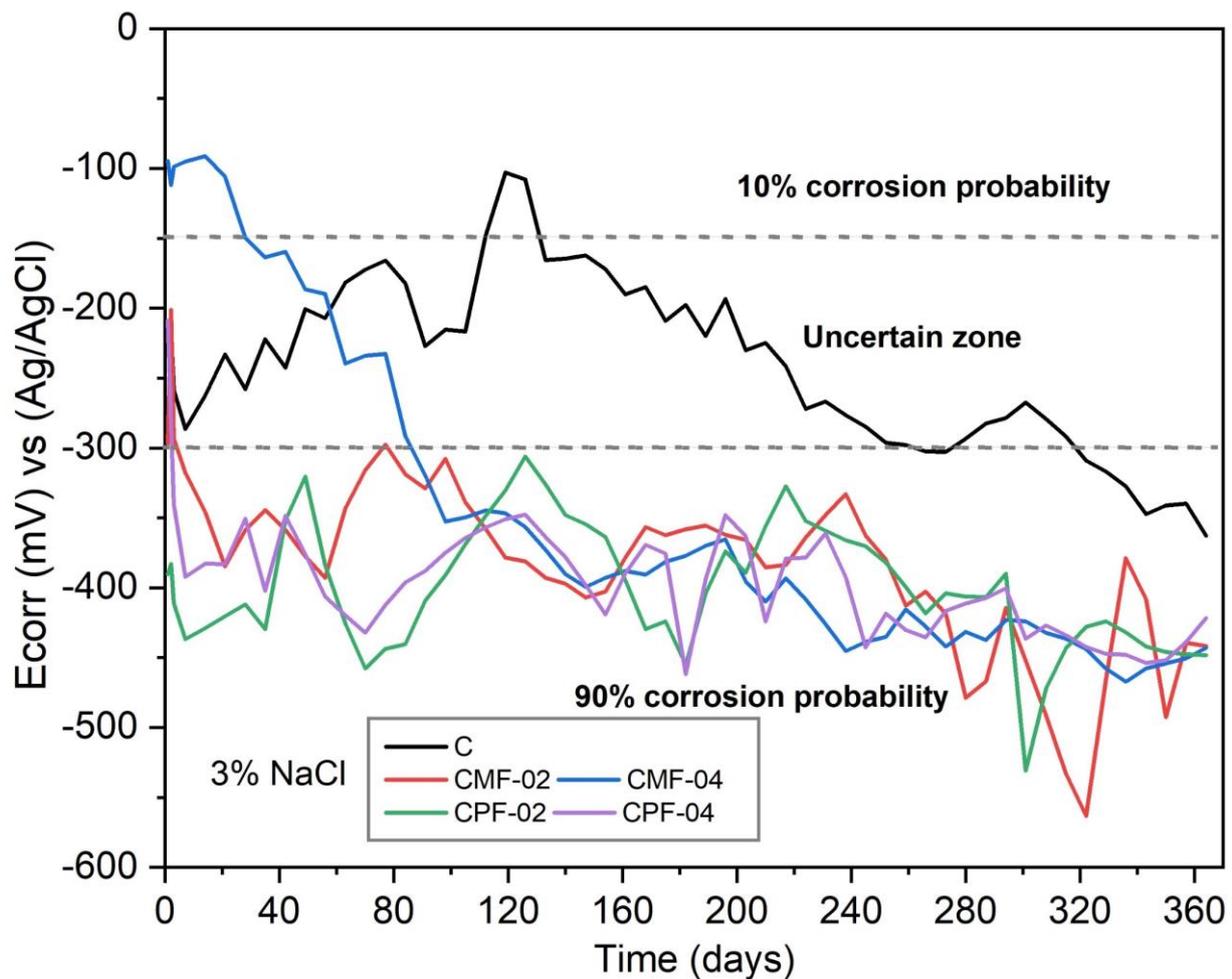


Figure 15. Corrosion potential values for structural steel

After 80 days the sample has a drop in  $E_{corr}$  potentials entering a zone of uncertainty, as time progresses and on day 365 of exposure this sample acquires more negative values from -400 mV to -550 mV entering a zone of 90% probability of corrosion, this fact could mean the increase of chloride ion content solution on the surface of the steel/concrete interface [76]. Samples CMF-02, CPF-02 and CPF-04 are in an active zone with 90% probability of corrosion from the beginning to the end of the measurement days, due to the high porosity of the samples, which causes higher voids in the cement paste and higher concentrations of oxygen, water and chloride ions [77, 78]. Chloride and oxygen ions compete to reach the surface of the reinforcing steel through a diffusion phenomenon [79]

#### 4.5. Interpretation of Electrochemical Noise Technique

The maintenance and repair of structural steel reinforced concrete for its safety needs effective monitoring and Inspection methods to evaluate the corrosion of steel reinforcement [80]. The aqueous chloride solution can quickly reach the embedded steel, so the onset of corrosion occurs in the metal [81]. Figures 16 and 17 show the results of Rn and the corrosion rate of concrete with treated fibers.

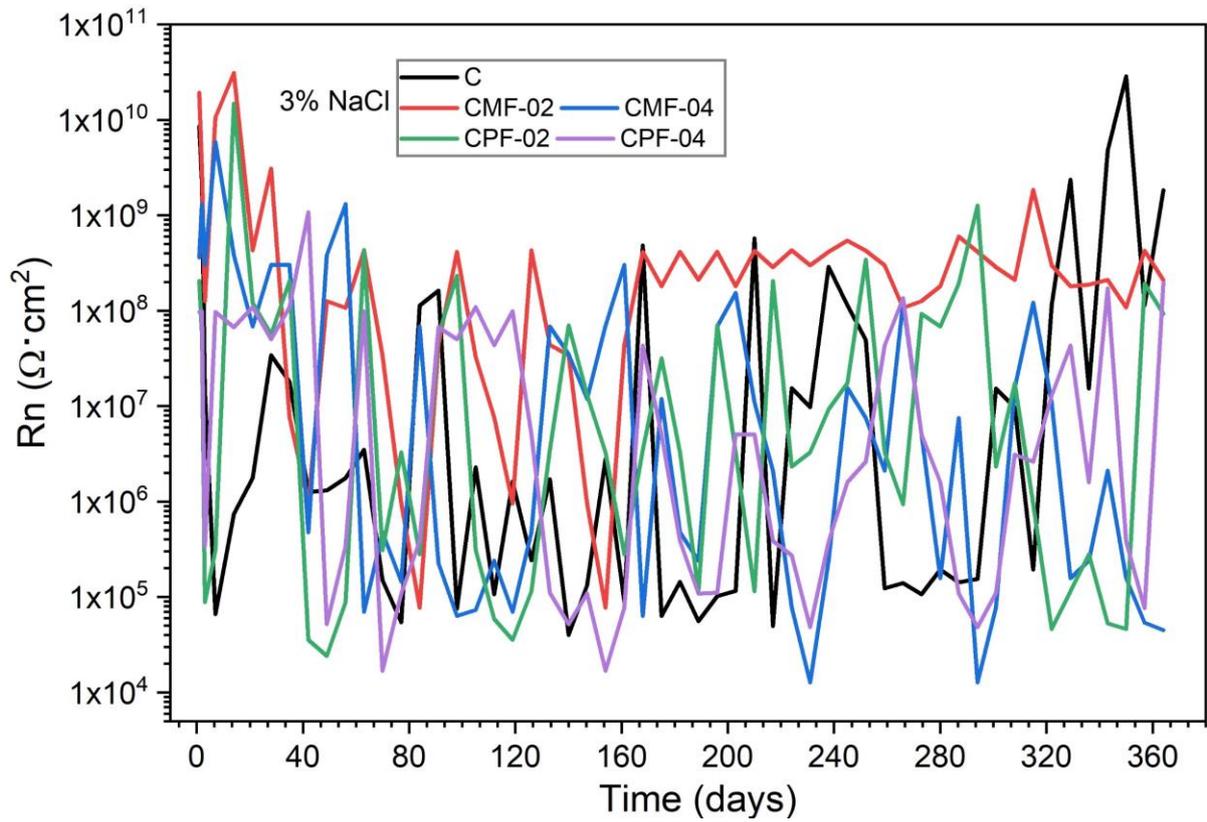


Figure 16. Rn values in the reinforced concrete

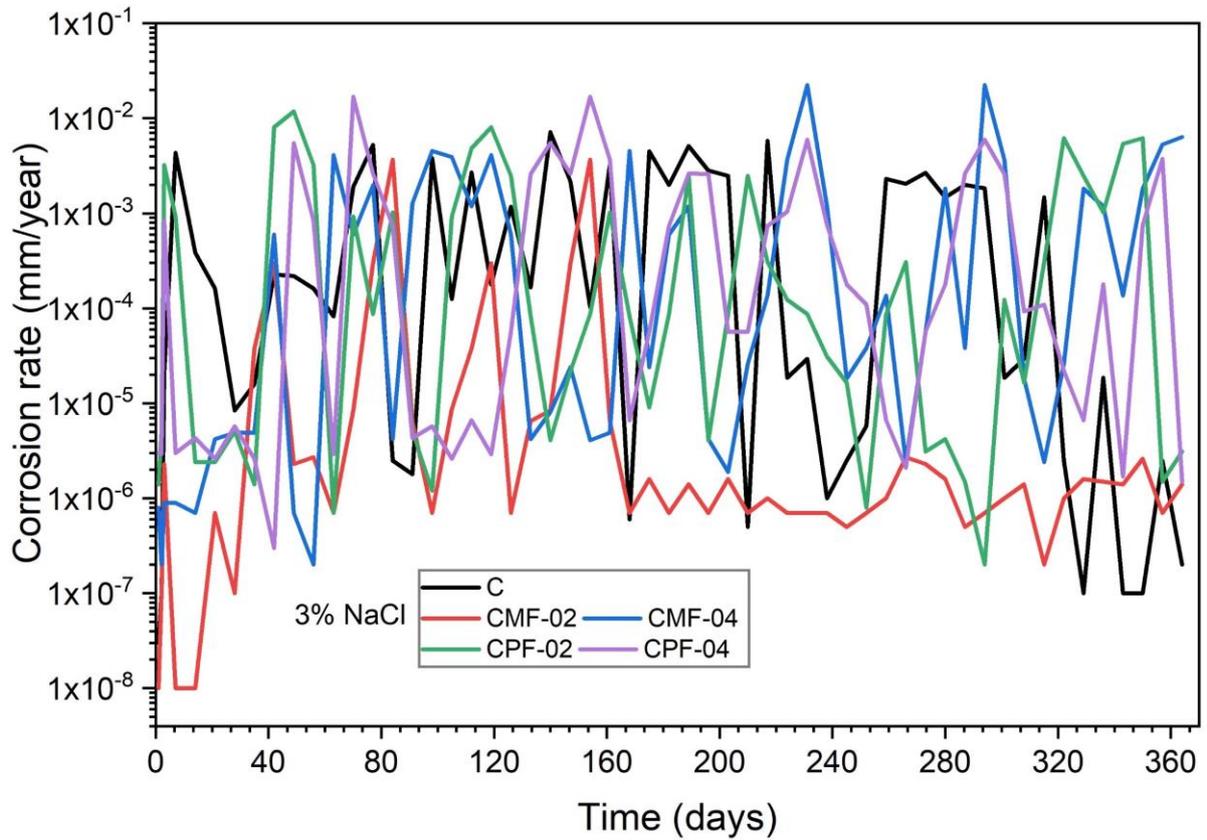


Figure 17. Corrosion rate for steel rod

On day 40 of monitoring, high and low transients of Rn with values between  $1 \times 10^{10} \Omega \cdot \text{cm}^2$  and  $1 \times 10^5 \Omega \cdot \text{cm}^2$  are observed for all samples, low values of Rn could indicate that the concentration of chloride ion increases on the surface of the steel bar, consequently, the corrosion rate is severe [82], i.e., the corrosion rate is inversely proportional to the values of Rn.

The values of UEO-treated samples are higher than the control sample, as the exposure time progresses the  $R_n$  values for natural fibers maintain the increase, high values of noise resistance, corrosion rate decreases, the entry of  $Cl^-$  in the concrete increases the rate by the reduction of the alkaline hydrated products of the cement that passivates the embedded steel [83], i.e., the steel bars are passivated and depassivated by the interaction of the aggressive agents that were transported through the pore network of the concrete.

At the end of exposure, all samples show high and low  $R_n$  values, these fluctuations possibly due to passivation depassivation events transient behavior. It is not clear if the trend would continue as low or if it would follow the same trend as the control. However, sample CMF-02 shows high and constant  $R_n$  values of  $1 \times 10^9 \Omega \cdot cm^2$  from day 160 to 365 days of exposure to the saline medium, this phenomenon may indicate that the treated fiber and the oil behave as a physical barrier, which may cause a slower diffusion of aggressive agents, also high  $R_n$  values indicate passivation in the metal and a negligible corrosion rate [46].

Quantitative information on the corrosion rate of steel is of utmost importance for the evaluation of repair methods, for service life prediction and for the structural evaluation of corroded elements [84].

An electrochemical parameter that can be obtained with the EN test is the type of corrosion that is present in the steel bars through the localization index results as shown in Figure 18. It has been argued in the literature that L.I., can be used to determine the nature of the type of corrosion with values approaching 1 being characteristic of localized corrosion [85].

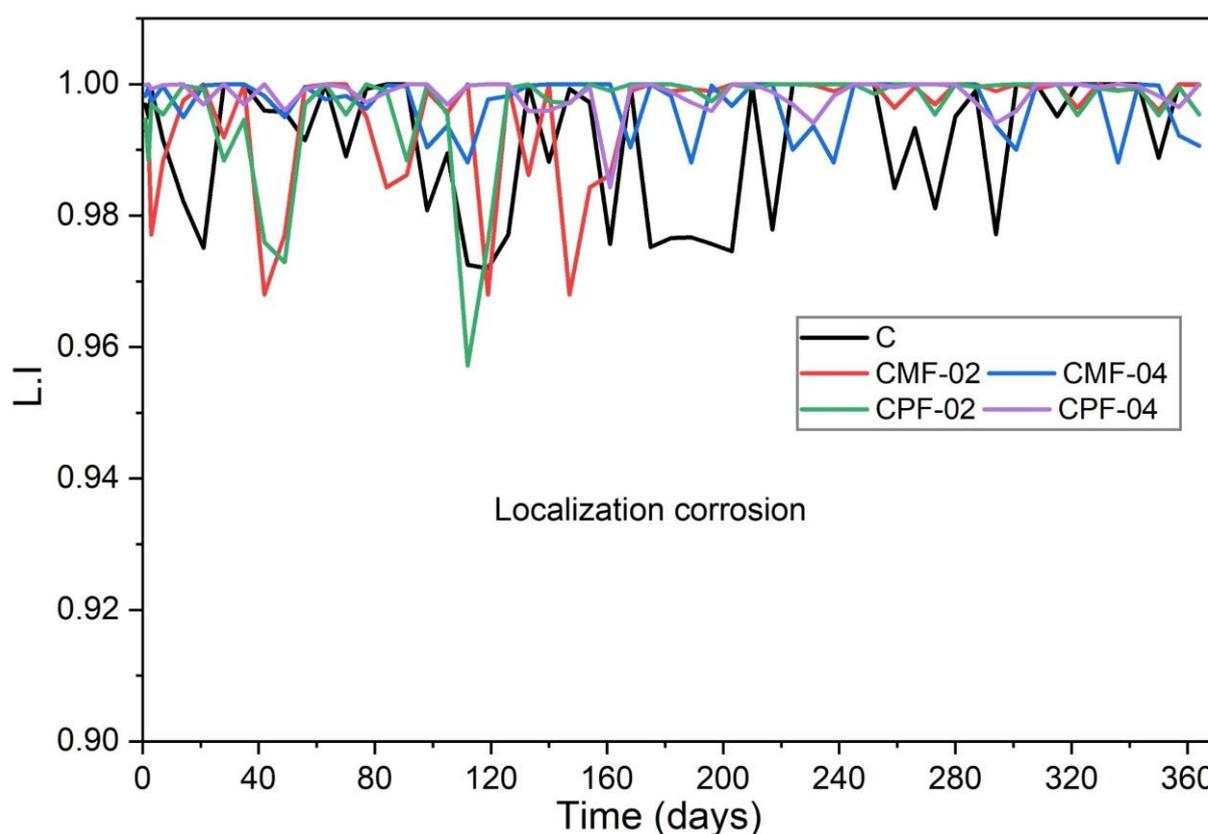


Figure 18. Type of corrosion in the steel/concrete system

It can be observed in a general way that all the samples with treated fibers have localized corrosion, that is, there are small areas of the rod surface, producing an important local reduction of the reinforcement section [64]. This type of corrosion is more likely to occur in reinforced concrete structures exposed to saline environments [86].

#### 4.6. Interpretation of Linear Polarization Resistance

The values of the treated fiber concrete samples can be observed in Figure 19. At the beginning, all samples acquired  $R_p$  values of around  $1 \times 10^5 \Omega \cdot cm^2$ , indicating that the steel is under low corrosion attack. As the immersion time progresses on days 210 and 365 of monitoring, sample CMF-02 presents a rise in  $R_p$  behaviors as observed with high values of between  $1 \times 10^5 \Omega \cdot cm^2$  and  $1 \times 10^7 \Omega \cdot cm^2$ , indicating a passivation in the metal surface and obtaining negligible corrosion damage as seen in Figure 20. Generally, steel corrosion is considered to be in the passive state if the current density is less than  $0.1 \text{ mA/cm}^2$  and in the active state for values greater than  $1 \text{ mA/cm}^2$  [44, 49]. The UEO has a positive effect on the corrosion rate by acting as a film or barrier around the metal; these results are comparable to those of other authors [87].

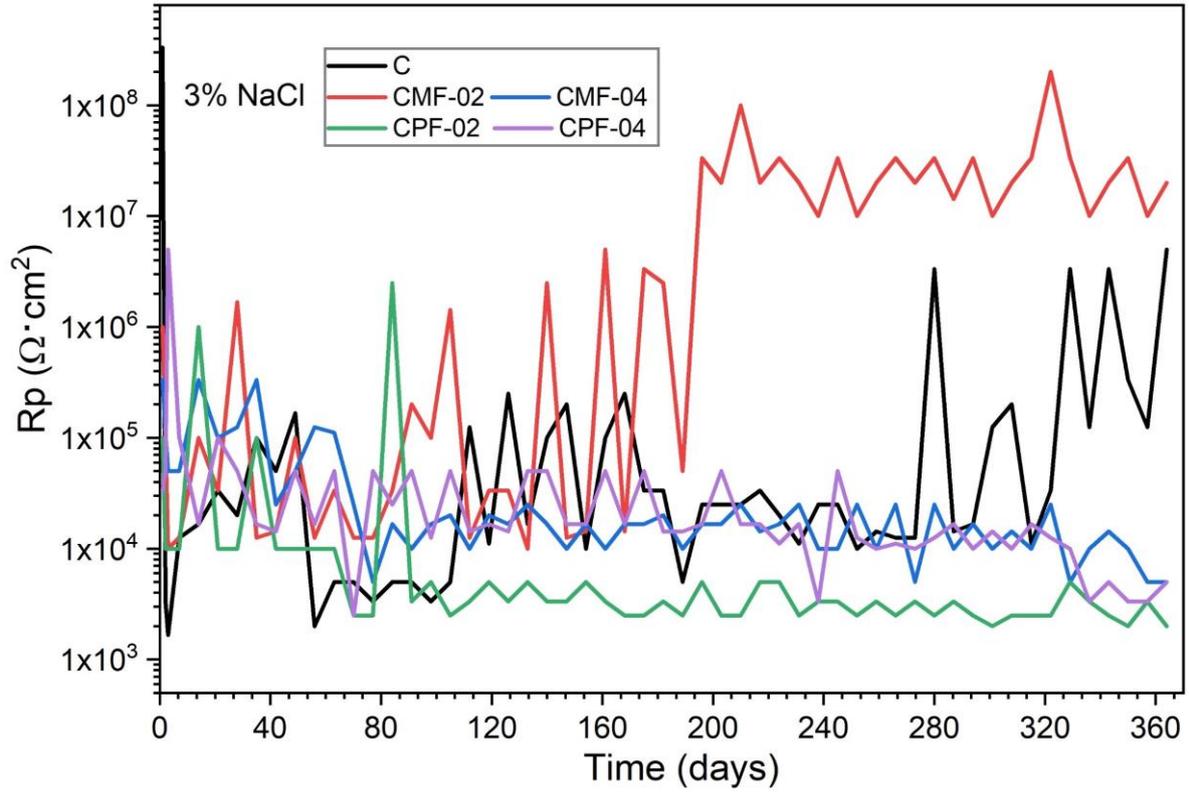


Figure 19. Average values of Rp

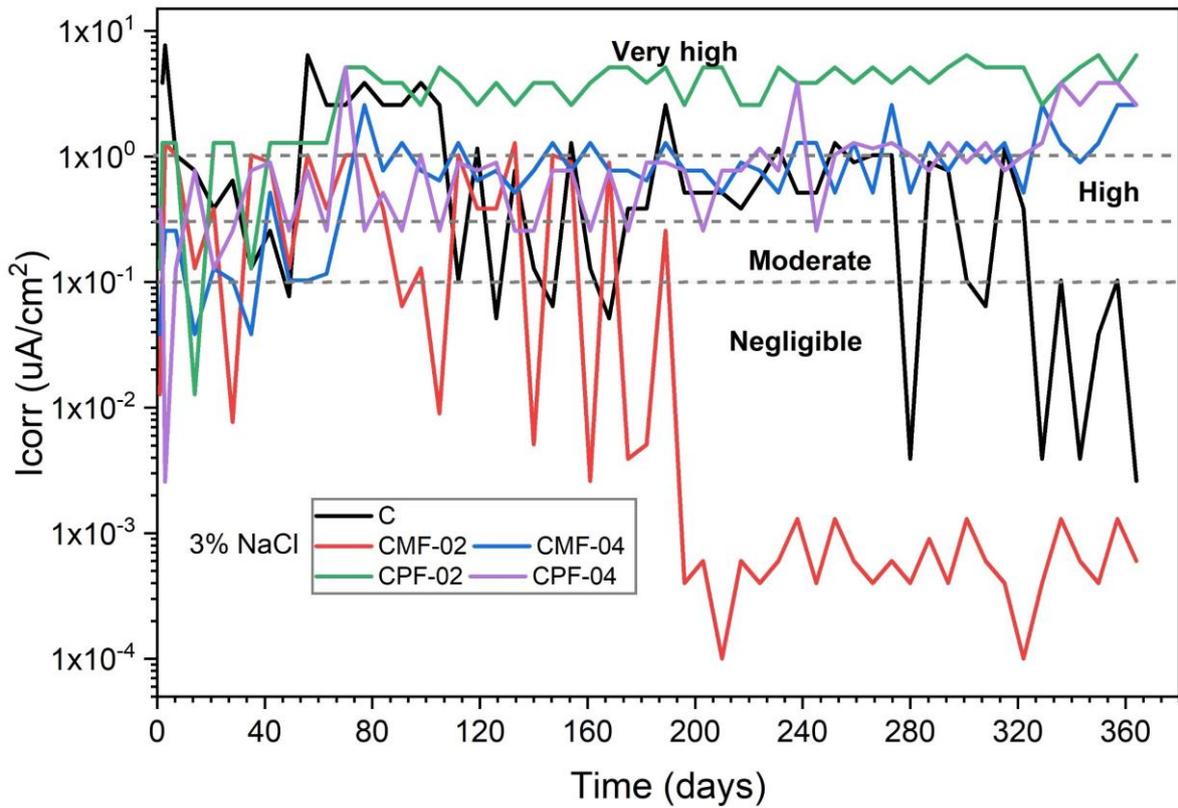


Figure 20. Current density values on the risk of steel corrosion

In the CPF-02 sample, low Rp values can be observed as the days of exposure progress, with values between  $1 \times 10^3 \Omega \cdot \text{cm}^2$  and  $1 \times 10^4 \Omega \cdot \text{cm}^2$  indicating high corrosion rate in the metal, due to poor adhesion of the fibers with the paste; likewise, the high porosity of the concrete paste by consequence has greater presence of oxygen diffusion and chloride

ions at the steel/paste interface, [88]. The values for the treated fibers have diverse from negligible to very high service life values compared to sample C. The samples CMF-04, CPF-02, and CPF-04 present very high  $I_{corr}$  values, increases in  $I_{corr}$  over time may not necessarily represent an increase in corrosion rate throughout the rebar, but rather the depassivation and initiation of active corrosion of greater amounts of rebar surface area [89]. Chloride penetration occurs mainly through capillary pores as free  $Cl^-$  chlorides by adsorption, diffusion, and capillary permeation [90]. The amount of fibers has a direct influence on the degree of corrosion of reinforcing bars in concrete [77].

### 4.7. Electrochemical Impedance Interpretations

The graph of real  $Z'$  on the abscissa and imaginary  $Z''$  on the ordinate were measured from high to low frequencies which are called Nyquist diagrams [80], these graphs present loops related to time constants originating from the structure and reactions at the electrode [91]. For the interpretation of the diagrams, the loops generated in the test are analyzed. Generally, for reinforced concrete corrosion, graphs present two or three loops (capacitive arcs with or without inductive loop), whose centers are below the real axis ( $Z_{real}$ ) and which are more or less separated [80, 91]. The high-frequency EIS spectra in the Nyquist diagram are usually a capacitive loop, which is generally attributed to the bulk concrete resistance representing  $R_e$  the ohmic resistance of the solution in the pores based on the electrode reaction time, and at middle to low frequencies it is possible to attribute the low-frequency loop to a charge transfer process in combination with a mass transport process ( $R_p$ ), while the straight line indicates a mechanism of ion diffusion through the pores [91-95].

The EIS results are shown in Figure 21, at the beginning of measurement at 24 hours, it can be widely observed that the control sample presents a better solution resistance  $R_e$  and a higher impedance  $R_p$ , with values of  $1 \times 10^6$  and  $1 \times 10^7 \Omega \cdot cm^2$  this phenomenon can be explained by the low porosity.

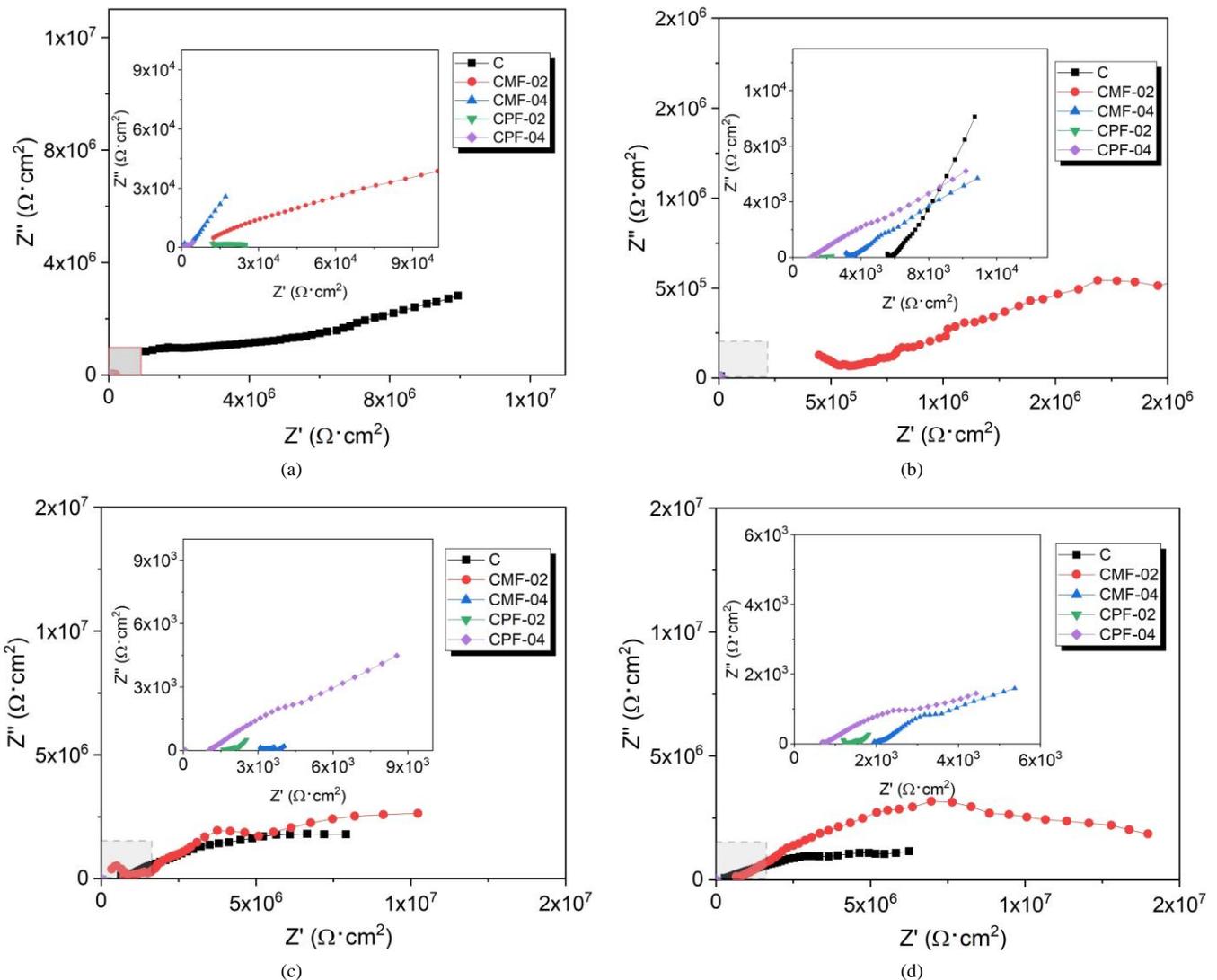


Figure 21. Nyquist diagrams for the days of a) 1, b) 168, c) 280, and d) 365

Small and depressed semicircles can be noticed for samples with treated fibers, indicating low  $R_e$  and impedance values due to the presence of larger pores in the concrete, causing not only increased diffusion of chloride  $Cl^-$  ions but also diffusion of  $O_2$  and  $H_2O$  [54].

For day 168, a remarkable improvement can be noticed for the fibers treated with used oil; the sample CMF-02 presents a better  $R_e$  as well as high impedance values of  $1 \times 10^6 \Omega \cdot cm^2$  with respect to the control sample. This fact could indicate the formation of a protective film of UEO on the metal surface by the high impedance values indicate a lower corrosion rate in the steel bar [96]. In addition, it was demonstrated that the physical properties of concrete, such as air content and porosity, have a direct relationship with corrosion damage to the metal due to the transport of species at the steel-concrete interface.

During the 240-day measurement, it was observed that the control sample had high values of  $R_e$  and  $R_p$  of  $1 \times 10^7 \Omega \cdot cm^2$ , which could indicate passivation in the metal. At the end of the 365 days of monitoring, it is appreciated that the sample CMF-02 has had a positive response to the corrosion process of the steel for 168 days, maintaining a constant impedance until the end of the exposure. High values in this low-frequency region may be associated with the presence of the passive film [97, 98].

## 5. Conclusions

The following conclusions can be drawn:

- High levels of workability were obtained in all samples incorporating treated fibers from impregnated UEO acting as water reduction and lubricant in cement particles, and aggregates.
- The CPF-02 and CPF-04 samples present 133% and 123% air content greater values, when compared with the control sample, ascribed to UEO high absorption in palm fibers, weakening the adhesion in fibers and cement matrix. As a consequence, the increase in voids in the cement paste porosity increased by 34% and 33%, respectively.
- The UEO impregnated effect in fibers modified the low-level properties of mechanical compression in concrete. The CMF-04 specimen exhibited higher resistance when compared with the other treated samples but was lower than the control sample.
- The  $f'f$  of all samples treated showed a decrease between 22% and 25% compared to the sample without added fibers. The UEO modified the fiber surface, losing the rough texture and promoting concrete specimens to present a fragile cracking failure.
- The low performance in mechanical properties may be due to poor adherence and cohesion in fiber paste materials to support external forces and stresses.
- All samples presented a 90% probability of corrosion, with more negative values of corrosion potential ranging from -400 mV to -550 mV.
- The sample CMF-02 presented a better positive performance in the corrosion rate on the steel with high values of  $R_n$  and  $R_p$  of  $1 \times 10^8 \Omega \cdot cm^2$ , indicating the formation of a protective film of UEO on the steel surface, decreasing the diffusion of  $Cl^-$ ,  $H_2O$ , and oxygen, and appreciating negligible corrosion damage on the road.
- In all samples, diffusion processes were observed on the steel/concrete interface, according to the EIS data.

## 6. Declarations

### 6.1. Author Contributions

Conceptualization, A.F.N.; methodology, A.F.N., J.U.C., and M.F.N.; investigation, A.F.N., E.C.M.C., M.F.N., and J.U.C.; data curation, A.F.N., O.A.G.N., C.A.G.P., and M.F.N.; writing—original draft, A.F.N.; writing—review and editing, A.F.N. and J.U.C.; supervision, J.U.C. and E.C.M.C. All authors have read and agreed to the published version of the manuscript.

### 6.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

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## 6.5. Conflicts of Interest

The authors declare no conflict of interest.

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